



Recyclability of novel energy harvesting and storage technologies for IoT and wireless sensor networks

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ABSTRACT

This paper aims to present a set of separation and recycling methods designed to recover valuable materials and components from innovative high-performance piezoelectric (PEG) and thermoelectric (TEG) generators and monolithic supercapacitors (SC) developed within the InComEss project. This project, a part of the European Horizon H2020 research program, focuses on creating environmentally friendly, cost-effective, and highly efficient Energy Harvesting Systems (EHSs) for powering wireless sensor networks. It combines advanced polymer-based composite materials for piezoelectric, thermoelectric, and supercapacitors, capable of harvesting and storing electrical energy from mechanical or waste heat sources. The authors initially identified key recoverable components. In PEG, these include Polyvinylidene fluoride (PVDF), silver electrodes, polyimide, fiberglass/resin composite and copper. In TEG the highest-value components are single walled carbon nanotubes (SWCNTs) and polymeric matrices, along with the SWCNTs, while in SC, aluminium, polyethylene terephthalate (PET) and carbon particles are targeted. This paper proposes a range of separation and recycling techniques involving mechanical, thermal, and chemical processes. These methods include microwave-assisted and pyrolysis thermal processes, various mechanical fragmentation and separation processes, hydrometallurgical recovery processes, and solvent-based dissolution methods. However, a comprehensive understanding of the end-of-life waste stream of these devices is necessary for further advancements. The authors are currently conducting a thorough laboratory assessment of the recovery methods to pinpoint the most effective recyclable options. The outcomes of this assessment will be shared in a subsequent article.

1. Introduction

The Internet of Thing concept and current demand for wireless sensor networks require the application of efficient devices with energy storage being key to their functionality. For this purpose, high-density, high-voltage lithium-ion batteries are generally employed. As a more sustainable alternative, devices such as supercapacitors (SCs) made from new materials such as polymer/carbon composites are emerging. Coupled with lightweight energy harvesting systems (EHSs), such as piezoelectric and thermoelectric devices that collect energy from the

environment, SCs eliminate the need for conventional batteries thus reducing costs and environmental impacts (Pappinisseri Puluckul and Weyn, 2022). Additionally, EHSs enable self-sufficient wireless sensor networks nodes operating indefinitely with an appropriate energy source. Promising devices combine energy harvesting and sensing, generating electrical energy from mechanical or thermal energy. However, popular piezoelectric and thermoelectric devices usually contain toxic lead-based materials and critical rare-earth elements (i.e., Bismuth), respectively (Bell and Deubzer, 2018; LeBlanc et al., 2014).

InComEss, an EU-funded project, investigates and designs alternative

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piezoelectric and thermoelectric materials (i.e., Polyvinylidene fluoride (PVDF), carbon nanotubes) for energy generation and supercapacitors for storage. In the project's initial stages, the project partners relied on their expertise and evaluated performance KPIs for specific use-case scenarios to select alternative materials in place of existing commercial state-of-the-art solutions. The manufacturing process of these innovative devices was then adjusted based on the attained results in relation to the anticipated KPIs. In line with circular economy principles, recycling of these devices is crucial to reduce their impact on material demand and resource depletion. Therefore, amongst the aims of InComEss is evaluating recycling technologies for obtaining high-value materials from recovered End-of-Life devices, which is the focal point of this paper.

This article outlines a circular approach for energy harvesting and storage devices developed within the project by assessing their recyclability and identifying valuable materials that can be recovered. First, the design and composition of the devices is assessed to identify technoeconomically recoverable materials. Then, the recoverable materials are proposed according to different parameters such as their cost, environmental impact associated to their extraction and processing, weight fraction in the devices and the likelihood to be recovered with high quality and recovery rate. Finally, for each device different recycling routes are discussed and proposed ensuring that potentially recyclable materials are not compromised.

2. Energy harvesting systems configuration and application

Energy Harvesting Systems (EHS) are used to power selected wireless sensor nodes (WSN) by the use of energy generators based on energy harvesting concept with the aim of implementing them in different IoT scenarios. On one hand, Fibre Optic Sensors (FOS) will be monitored for Structural Health Monitoring purposes in buildings and aircrafts by using a miniaturized FOS interrogator with low power consumption specifically constructed for energy harvesting) while on the other hand, wireless temperature sensors and MEMS (Micro Electro-Mechanical System) sensors will be monitored in vehicles. The EHS described in this paper is composed by energy generators such as **Piezoelectric Generator (PEG)** composed by lead-free piezoelectric composite strips/flat tapes based on PVDF and **Thermoelectric Generator (TEG)** based on innovative high-performance thermoplastic-based p- and n-type thermoelectric composites (PEEK) together with a monolithic **super-capacitor (SC)** developed with a printable high energy density PANI/carbon-based composite electrode to store energy harvested by the

generators. The full concept is depicted in Fig. 1. A power conditioning circuit connect the energy generators with the energy storage element powering the sensor and the transmission of data through the IoT platform. The power conditioning circuit is based on electronic components available on the market and its recyclability is not considered in this paper.

3. Design of EHS devices and Bill of Materials in InComEss project

In this section, the Bill of Material (BOM) of the devices is described as well as their design, which will serve as a basis for the study of the recycling techniques.

3.1. Piezoelectric generator (PEG) design and composition

PVDF is used as the main polymer for piezoelectric composites. PVDF is a thermoplastic polymer which is blended with a dispersing agent (organic additive). An extrusion-based technology is used for the manufacturing process of the strips as it is suitable for PVDF orientation. In this way, the polymer pellets are fed into an extruder consisting of a twin screw and a barrel for melting employing heat, and then, the molten polymer is forced through the die and transformed into a long strip while it is quenched in water, becoming solid. In a second process, the strips are stretched to increase the crystalline beta-phase of PVDF and therefore, enhancing piezoelectric properties. Silver ink is printed as electrodes on both sides (top and bottom electrodes) which is needed for their polarization (application of high electrical field through the thickness). Once poled, the piezoelectric strips are cut according to the final design and bonded onto a fiberglass composite carrier beam using an epoxy resin as adhesive. Silver-based contacts are connected to the encapsulation layer through the copper electrodes, and the boards are glued with epoxy resin at the bottom and upper sides of the piezoelectric strip. In the final design the copper will be printed on the encapsulation layer which is made of polyimide (kapton). Then, using epoxy resin this device adheres to the cantilever made of fiberglass composite. Two electrodes connect the strip's bottom and upper part using two wires. The piezoelectric generator prototype based on PVDF can be seen in Fig. 2.

3.2. Thermoelectric generator (TEG) design and composition

The InComEss project pursues to develop innovative high-

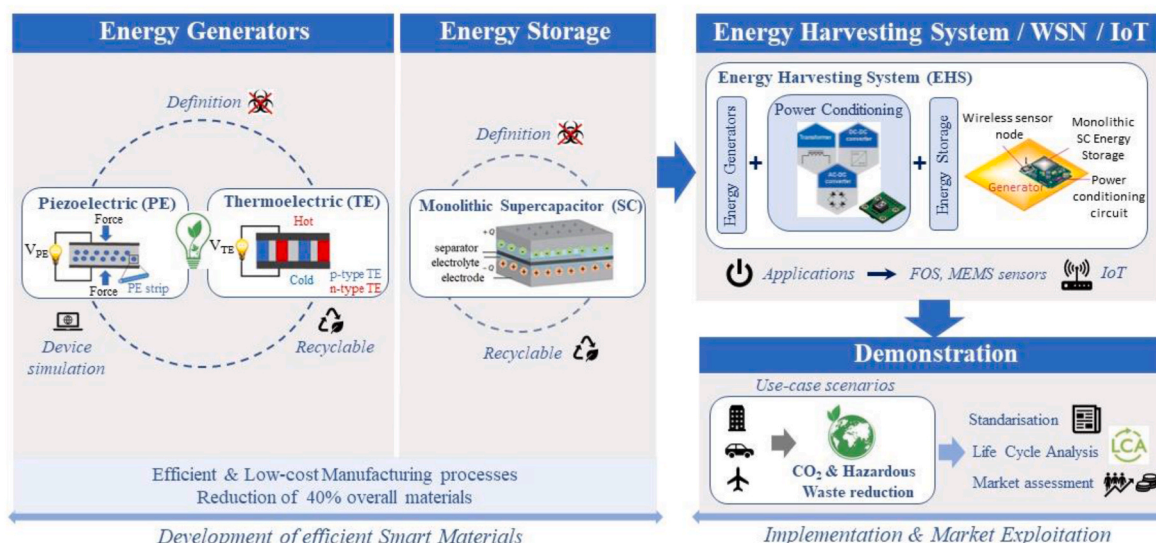


Fig. 1. InComEss concept description.

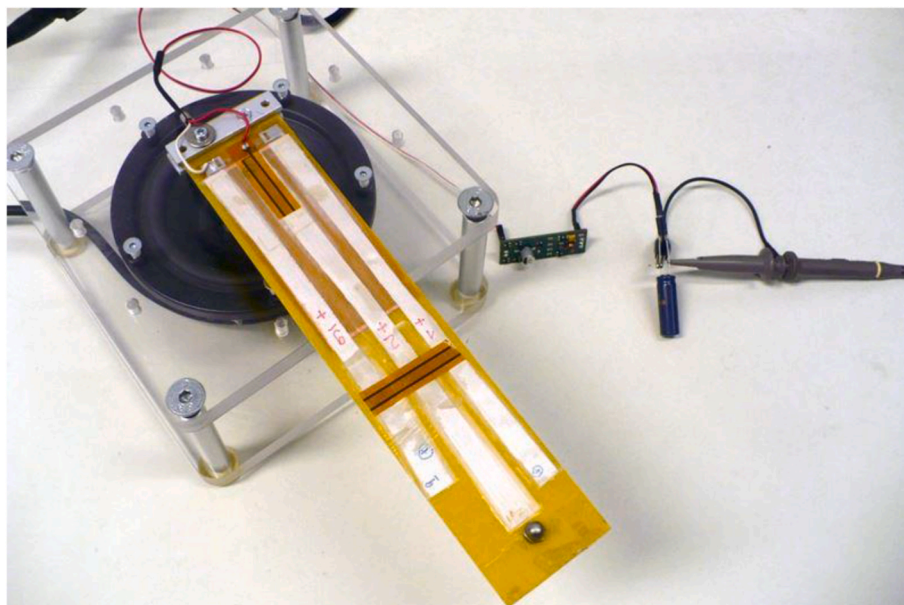


Fig. 2. Piezoelectric generator (PEG) based on PVDF tapes.

performance thermoplastic based TE composites with enhanced Seebeck coefficients for their integration into a thermoelectric generator for applications requiring a high temperature range (from $-25\text{ }^{\circ}\text{C}$ up to $+240\text{ }^{\circ}\text{C}$). Consequently, the polymer selected and used as matrix for the development of these thermoelectric materials is poly(ether ether ketone) (PEEK) a high-temperature performance thermoplastic polymer. The thermoelectric generator design is based on a combination of materials with p-type character (majority of the charge carriers are electrons) and n-type (holes are the dominant charge carriers). The thermoelectric composites are essentially produced by the environmentally friendly melt compounding method in which the polymer matrix is melted and single walled carbon nanotubes (SWCNTs) as conductive fillers are incorporated under shearing action in the molten matrix at concentrations lower than 3 wt% for the case of p-type composites. To obtain n-type composites, polyethylene glycol is used as a switching additive (at concentrations around 15 wt%) to induce a negative Seebeck coefficient (switching from p-to n-type). As a result of the melt-mixing process, composite strands are obtained. These strands are then formed into sheets by the hot compression molding process. According to the final design of the generator, plates of p- and n-type PEEK composites are compression moulded and strips of the required dimensions forming the legs are then cut therefrom. More explanations regarding the material production process are given in (Krause and Pötschke, 2022). An example for a prototype of a generator (shown for

the polymer type polypropylene (PP) and its performance can be seen in (Doraghi et al., 2023; Luo et al., 2017). P- and n-type legs are connected by sputtering with copper foil or conductive silver paste. In the case of a block design as used in InComEss, this assembly was embedded in an epoxy resin to stabilise the geometry (see Fig. 3). This design allows the TEG to be exposed to cold on one surface and heat on the other. Fig. 3 shows one example of the TEG prototype.

3.3. Supercapacitor (SC) design and composition

The preliminary design of the InComEss project's printed monolithic supercapacitor is represented in Fig. 4A. Description of its manufacturing methods and typical electrical performance can be found in (Arvani et al., 2020; Keskinen et al., 2018; Pourkheirollah et al., 2023). PET/aluminium laminate has been used as substrate for the construction of the supercapacitor. The total thickness of the substrate and encapsulation (element 1) will be $60\text{ }\mu\text{m}$, with the thicknesses of the individual layers being PET $50\text{ }\mu\text{m}$ and aluminium $10\text{ }\mu\text{m}$. In this structure aluminium acts as a barrier layer. The current collector (element 2) will be made from Henkel Electrodag PF407C conductive ink, a graphite filled composite that is cured at high temperatures ($90\text{ }^{\circ}\text{C}$ – $120\text{ }^{\circ}\text{C}$). The electrode (element 3) will consist of a water-based PANI/carbon ink, made by mixing chitosan binder with active material powder composed of PANI/carbon composite synthesized through

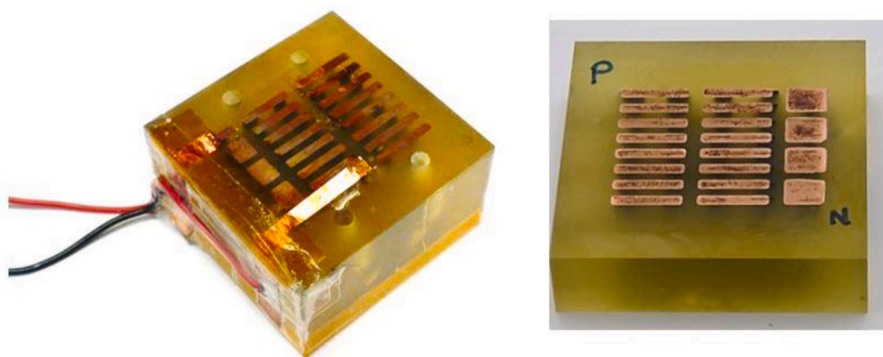


Fig. 3. Example of a thermoelectric generator (TEG) developed in InComEss.

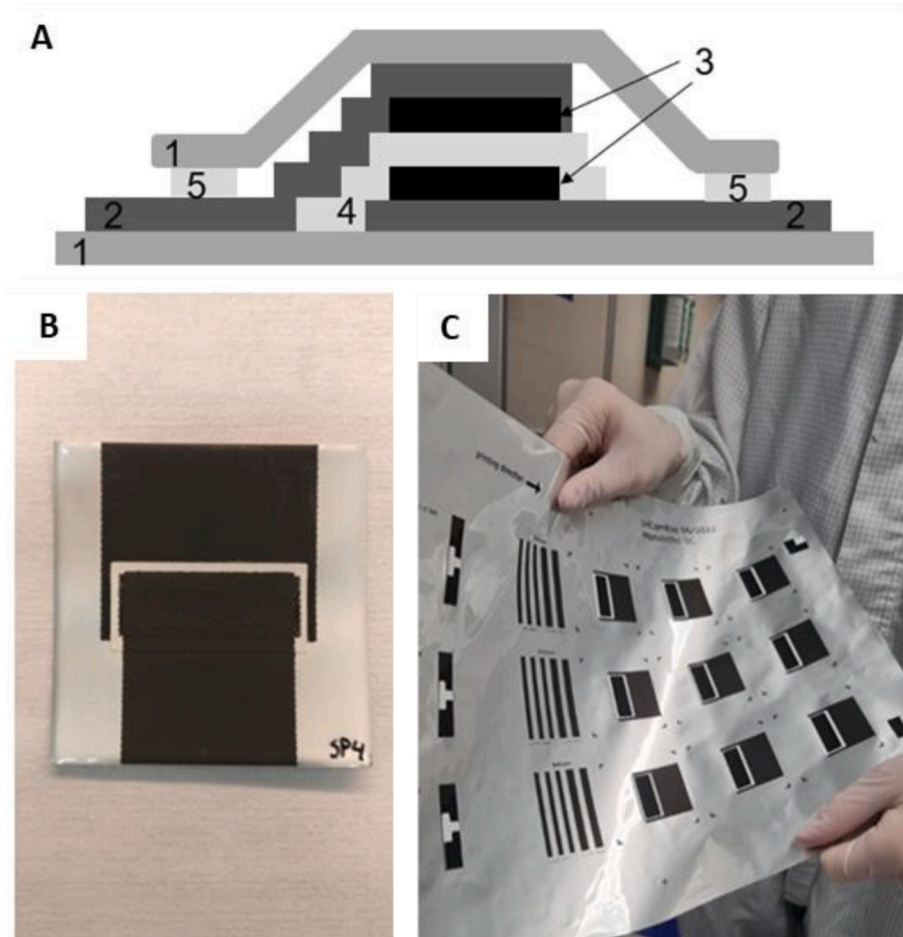


Fig. 4. A. Schematic structure of the SC: 1. Al PET (substrate + encapsulation), 2. Current collector, 3. Electrode, 4. Separator, 5. Adhesive. B. Single screen-printed SC. C. Sheet of the screen-printed SCs and test patterns.

in-situ chemical oxidation polymerization of aniline and carbon with ammonium persulfate (APS) as oxidant (Kong et al., 2008; X. Zhao et al., 2019). The separator (element 4) will be a talc-cellulose-chitosan (20:5:1 mass ratio) mixture. Finally, the electrolyte will be a 0.5 M Na_2SO_4 electrolyte. To ensure the sealing of the device within the encapsulant layers, the 3M 468 MP acrylic adhesive is used around the electrode area to seal the device.

The final SC prototype can be seen in Fig. 4B, as well as the sheet of the multiple SC made by the screen-printing technique (Fig. 4C). The weight fraction of all components is provided in Table 1.

Table 1
Supercapacitor composition.

Component	Material	Weight percentage (%)
Encapsulation & substrate layers	Aluminium	20.5%
	PET	53.0%
Current collector	Graphite particle	3.3%
	Thermoplastic matrix	2.2%
Electrode	Activated carbon particle	0.2%
	Chitosan/PANI matrix	2.1%
Separator	Talc	2.8%
	Cellulose	0.7%
	Chitosan	0.1%
Adhesive	3M 468 MP tape	11.6%
Electrolyte	0.5 M Na_2SO_4	3.5%

4. Consideration on the devices end of life

The life cycle of a product comprises the stages between design and eventual disposal, including production and maintenance. In a Circular Economy context, the definition of “end-of-life” (EoL) is aimed at minimizing waste and maximizing resource utilization by promoting a closed cycle where products, materials and resources are reused, repaired, remanufactured, and recycled to extend their life cycles. The length of each product’s life cycle depends on several factors including product type, market demand and technological innovation. The design of an integrated EHS should consider the separability and reparability of its devices (i.e., PEG, TEG, and monolithic SCs), both from the system and its application.

Separability, understood as the possibility of separating the device from the EHS or its application and recovering it for reuse, repair, or recycling, is crucial for EoL assessment. Combined with the potential for reparability, separability plays a vital role in reducing waste and conserving resources, thus promoting the circular economy and contributing to the sustainability of the device and the system. In addition, the separability of the EHS and devices is an important factor that will determine the expected lifetime of the device itself. If the device is not detachable, for example if it is integrated, the EoL of the application and its waste stream will apply. This would mean that the EoL of the device is determined by that of the application. For example, if considering a potential automotive application for a light vehicle, a service life of about 10–15 years on average is expected (Buberger et al., 2022), while for applications in the construction sector, such as buildings, the expected service life is much higher at 50–70 years (Feng et al.,

2022), and in the aeronautical sector an aircraft is estimated to have a service life of 20–30 years (Pinheiro Melo et al., 2020).

On the other hand, if the device is removable, which is considered the preferred option, its EoL is determined by its own life cycle producing a waste stream independent from that of its application. Therefore, in order to minimize waste, it is important to design the EHS taking into account, in addition to the BOMs, the separability and repairability its devices recoverability, and the recyclability of valuable materials which will determine the sustainability of the device throughout its useful life.

4.1. Expected waste streams and recycling route

Despite the challenges hindering the application of reverse logistics, the relevance of incorporating it in the lifecycle management of TEG, PEG, and SC devices cannot be overlooked. While it is anticipated that these devices will likely end up in the Waste Electrical and Electronic Equipment (WEEE) stream due to their characteristics and potential detachability, their novelty and expected low volumes make it premature to envision a dedicated take-back scheme or infrastructure for reverse logistics processes. Nonetheless, the short-term use of these devices may provide a unique opportunity to test and refine reverse logistics strategies. It is worth noting that established industrial WEEE treatment facilities may not be optimally equipped to handle the recovery of valuable materials from the innovative TEG, PEG, and SC devices developed in the InComEss project. Consequently, despite these hurdles, exploring reverse logistics and reparability in the context of these devices is crucial for developing effective and sustainable end-of-life management solutions. The constraints of WEEE recycling processes are the result of the structural nature of the devices, which contains materials not commonly found in WEEE. Thus, polymeric compounds such as PVDF, PEEK and Kapton®, as well as other materials of high interest and value such as nanocarbon tubes and particles, cannot be recovered by the technologies applied in WEEE recycling. Consequentially, recycling of these devices may drive the search for the best recycling techniques towards the recycling of polymeric matrixes comprising mechanical and thermal (pyrolysis, fluidized bed pyrolysis and microwave pyrolysis) and chemical-based approaches (solvolysis) (Krauklis et al., 2021).

5. Research the different recycling techniques

Before getting into the different recycling techniques for the EHS devices, each device is evaluated according to its design to define which high-value materials could be recovered from them. A systematic literature review (Xiao and Watson, 2019) has been done to find out the available recycling techniques to recover these materials from the different components of each device.

5.1. Recoverable materials

This section explains which are the different recoverable materials that can be obtained from each device. The overall goal is to reach the highest recovery rate, although it is explained here why some materials are more interesting than others.

5.1.1. Piezoelectric generator

Considering the PVDF piezoelectric strips bonded to the surface of the cantilever of the PEG can be removed from the devices without damage, the products to recover would include PVDF, together with the silver electrodes that are joined to the PVDF strip in the cantilever design, polyimide (from Kapton®), fiberglass/resin composite and copper. It is important to clearly define the separation way of the PE strips from the cantilever to extract the PVDF in good conditions. In this way, PVDF can be directly reused in other PEG, avoiding a new production. For the rest of the materials, the most interesting ones are the

silver and the copper due to their cost and environmental impact associated to their extraction and processing (Farjana et al., 2019; Yang et al., 2022).

5.1.2. Thermoelectric generator

TEGs are primarily made up of polymeric matrices, with PEEK being a thermoplastic known for its exceptional properties. PEEK is chosen for its high thermal stability (up to 240 °C) required for the application. It acts as a host for SWCNTs and polyethylene glycol in the thermoplastic nanocomposites. While the ideal recycling efficiency for TEGs is 100%, SWCNTs hold the highest value in the device, contributing to over 90% of the costs despite their small percentage. According to the market, SWCNT (3000€/kg) (Ossila Ltd, 2023) shows the highest cost per kilogram in comparison to PEEK (52 €/kg) or polyethylene glycol (60€/kg) (PlasticFinder s.r.l, 2023). Recovering SWCNTs during recycling is challenging due to their complex isolation from the polymeric matrices. For this reason, the recoverable materials from TEGs would be the polymeric matrices, along with the SWCNTs, that can be reused for producing new TEG components or other applications.

5.1.3. Supercapacitor

The most valuable material from the substrate and encapsulation layer is aluminium although improved device design in the future could reduce the foil thickness from the current 10 µm to 100 nm, making it difficult and potentially unsuitable for recycling. Aluminium is a critical raw material (Ippel, 2023) that is widely used in various industries. Additionally, found in the substrate and encapsulation layer, PET represents more than half of the weight of the SC, and its recovery and valorisation should also be considered. Recycled PET may not be easily introduced into food packaging due to concerns about residues or contamination, but it may be valuable and applicable in the textile industry (Mpika et al., 2023). In the case of, electrode and current collectors contain carbon particles, their recovery and reuse can be considered although their low weight fraction makes it challenging to achieve a reasonable recovery rate. Na₂SO₄ is a common by-product of several industrial processes and is often considered a hazardous waste (Zhu et al., 2022, p.). The talc, main component of the separator element is a cheap (Statista, 2022) and common material so its recovery is not considered. PANI and Chitosan are also not considered as recoverable material due to their low weight percentage and low economic value. The Acrylic adhesive makes up about 1/3 of the weight of the SC, but as it is classified as a Group 7 plastic,¹ it can be difficult to recycle.

5.2. Recycling techniques

This section explains the recycling techniques available for each device based on the recovery of the materials above identified.

5.2.1. Piezoelectric generator

5.2.1.1. *Degradation of the encapsulation layer.* Epoxy resin is a type of thermosetting plastic difficult to melt and reshape. However, there are mechanical, thermal, and chemical techniques that can be used for its recycling. Chemical processes present an important challenge considering the potential effect on other target components. Mechanical recycling by crushing would result in a mix of crushed composites that could be used as reinforcement pieces (Z. Tian et al., 2022). Epoxy resins may be removed by combining heat and pressure exposing the material to temperatures above its softening point. Here, the adhesive is softened, and the components can be de-bonded. Temperatures of about 400 °C or higher, will degrade the resin allowing for component separation as well. However, the integrity of the PVDF strips may be affected

¹ ASTM D7611-20: Resin Identification Code Standard.

hindering their recycling. It is proposed to treat the encapsulation layer by microwave assisted degradation with glacial acetic acid and AlCl_3 as catalyst (Y. Wang et al., 2015). Aiming at a sustainable method, both the acetic acid and the AlCl_3 could be recovered and reused, potentially resulting in lower treatment costs. This method would allow to decompose the epoxy resin and release the piezoelectric strips. The use of an organic solvent such as NMP (N-methyl pyrrolidone) in a microwave assisted degradation at 80 °C would also be an option (F. Tian et al., 2020), however, it is not clear how this treatment would affect the physicochemical properties of the PVDF strips. Furthermore, NMP is persistent in the environment and toxic to humans and aquatic life deeming it a non-environmentally sound option to consider.

The boards composed of fiberglass and epoxy resin may also be recovered whole and destined for reuse in the production of the same electronic components, allowing to improve the circularity of the device.

Once the encapsulation layer has been degraded, the main recoverable materials from PEGs include: PVDF, Polyimide, silver, and copper. The available treatments to target each of these materials are described below.

5.2.1.2. PVDF. Recycling of fluoroplastics such as PVDF is economically attractive due to their high stability and value. In general, recycling of PVDF has been studied and reported in the framework of LIBs (Lithium-Ion Batteries) treatment where it is found as a polymeric binder, evidencing further research is needed on PVDF recycling retrieved from other waste streams (Bai et al., 2020; Fu et al., 2021; Laali et al., 2018; Wu et al., 2022; Yu et al., 2022).

Recycling PVDF can be a challenge due to its high resistance to chemical and mechanical degradation processes. Different recycling techniques are being researched:

Chemical treatment: The low chemical reactivity of PVDF challenges solvolysis. (Marshall et al., 2021) presents a summary of effective solvents and diluents for PVDF recovery including acetone, DMF (dimethylformamide), DMP (dimethyl phthalate) and supercritical fluids. In (Sarkar et al., 2021), the use of organic solvents including DMF, NMP (N-methyl pyrrolidone) and THF (tetrahydrofuran) was evaluated. Addressing the use of greener solvents, a study by (Bai et al., 2020) evaluated the use of a bioderived solvent Cyrene® (dihydrolevoglucosenone) which proved to be effective at temperatures higher than 80 °C allowing for PVDF recovery by precipitation at lower temperatures.

The use of supercritical CO_2 combined with DMSO (dimethyl sulfide) as a cosolvent is reported in (Fu et al., 2021) having achieved a 98.5%wt recovery of pure PVDF from LIBs cathode in 13 min under a pressure of 80 bar at 70 °C. Other solvents studied include fatty acid methyl esters (FAMES) and ionic liquids (Parga et al., 2005; M. Wang et al., 2020), however, these are costly and therefore an economically unviable option. New technologies for PVDF degradation include the combined use of molten salts and high voltage discharges.

Thermal treatment: At room temperature PVDF does not easily corrode in the presence of acids, strong oxidizers or even halogens. Additionally, suitable solvents are hazardous thus the development of thermal processes has been evaluated.

The major challenge on recovering PVDF by thermal decomposition is the production of fluorinated organic compounds such as fluorobenzene and HF (hydrogen fluoride) (Laali et al., 2018), which could lead to environmental pollution. Direct calcination can result in the emission of these pollutants being pyrolysis in a completely closed environment a better processing option with full control and possibility of treatment of the exhaust. (M. Wang et al., 2023) reports an optimum pyrolysis temperature range between 500 and 580 °C.

Under moderate alkalinity PVDF can be recovered without undergoing defluorination or physical deterioration. The use of ammonia has been shown to be effective in eliminating dioxin generation in high temperature dehalogenations. (Morita et al., 2023) proved PVDF did not

deteriorate in a process carried out at 100 °C, for 2 h at 10M NaOH. However, the high corrosivity of inorganic fluoride directly hinders the lifetime of the equipment increasing costs.

Mechanical treatment: PVDF can be recycled as a thermoplastic through extrusion re-melting. Despite scarcity of reports on the use of mechanically recycled PVDF, (Veiga et al., 2020) compared a reference sample to two recycled specimens/samples. They concluded no major changes of PVDF properties were observed after extrusion rendering mechanical treatment as a viable recycling option. Further studies are reported in (Wahlström et al., 2021).

5.2.1.3. Polyimide (Kapton®). The recyclability by reuse of polyimide thermoplastics such as Kapton® is between 5 and 8%, leaving the remaining 92% as an unclear problem to solve.

Thermal treatment: Oxidative degradation of polyimides (PIs) starts at 350 °C. Subjected to thermal degradation, Kapton® generates mainly CO_2 , CO and other volatiles. Pyrolysis can be used to produce reticulated structures such as microporous membranes to applied as gas filters (Rusu and Abadie, 2020).

Chemical treatment: Hydrolytic degradation and water-induced plasticization is an environmentally sound method, but highly dependent on time (month scale) and energy as it requires thermal activation, making energy and cost-inefficient. Chemical treatment has been combined with mechanical processes to achieve more effective and efficient recycling. For instance, the advantages of ball milling have been recently demonstrated by (Olifirov et al., 2013). Ball milling is a simple process, economically viable and perceived as a greener option to chemical recycling. Here, high purity starting monomers and partially imidized powders are obtained in a metastable state.

Mechanical treatment: Products such as Kapton® can also be reused. For instance, applying ion etching, (Choi et al., 2019) developed patterned through-holes on the film which combined with the intrinsic electrostatic forces from Kapton® provide the capacity to efficiently capture particulate matter ranging between 0.3 μm to 10 μm in long-term dust filtration.

5.2.1.4. Metals: silver (Ag) and copper (Cu). In PEGs the polyimide component contains copper, therefore, the method selected for recovering this metal will depend on that selected for recycling the PI. Consequently, Cu may need to be recovered from pyrolysis ashes or by acid leaching of recovered PI. It may also be necessary to evaluate the effect of Cu on the product obtained from Kapton® recycling considering its future use.

Ag electrodes are soldered to PVDF strips thus the recovery of Ag will depend on the treatment chosen to recover PVDF. Silver based contacts could be recovered manually adding economic benefit to the overall PEG recycling process.

The Ag-based ink used for printing the PVDF strips requires further research to determine how it interacts with the selected method and products.

Table 2 shows a summary of the possible recycling techniques for the PEG:

A proposed sequence of techniques for the recycling of PEGs is presented in Fig. 5.

5.2.2. Thermoelectric generator

5.2.2.1. Attack to the sputtered copper connections. Copper sputtered connections in the TEG can be recovered through recycling techniques such as manual sorting, mechanical processing, pyrometallurgical recycling, hydrometallurgical recycling, and electrolytic recycling. Currently, approximately 80% of copper from waste is collected for recycling in Western Europe (Henckens and Worrell, 2020). In the case of the TEG, the copper sputter connections can be separated by subjecting them to a hydrometallurgical treatment using nitric acid (3M) at

Table 2
Recycling techniques selected for PEGs.

Components	Composition	Recycling techniques	Recoverable materials	Reference
Encapsulation layer	Epoxy resin + Fiberglass Polyimide + printed copper	Microwave assisted degradation with Tartaric acid H ₂ O ₂	Piezoelectric strips	Zabihi et al. (2020)
		Milling in high-energy ball mill	Polyimide powder	Olifirov et al. (2013)
		Pyrolysis at T > 300 °C	CO ₂ together with CO and other volatiles	Rusu and Abadie (2020)
PE film	PVDF/BYK	Dissolution: a. Propylene Carbonate b. Dihydrolevoglucosenone	Membrane casting Gel formation	a. (Nishiyama et al., 2016) b. (Marino et al., 2019)
Electrodes	Silver	Dependent on treatment chosen to recover PVDF. Silver based contacts: manual recovery. Silver based ink: further research required	Silver contacts	–
	Copper	Dependent on method selected for recycling the PI	Copper compounds	–

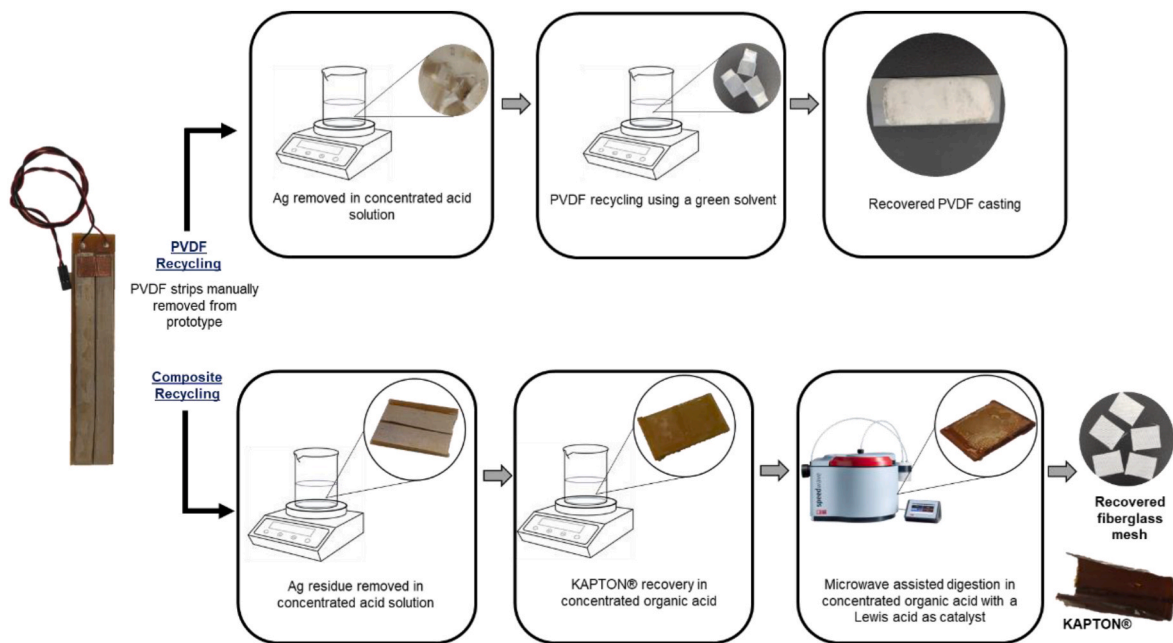


Fig. 5. Sequence of techniques for the recycling of PEGs.

60 °C for 5 h and a pulp density of 25 g/L (M. Kumar et al., 2014). The copper can then be extracted as copper hydroxide (II) by adjusting the solutions pH to 6 using a 0.5 M sodium hydroxide solution (Balintova and Petrilkova, 2011). In comparison with other recycling methods, such as pyrometallurgy, the hydrometallurgical method based on the leaching with nitric acid among others seems to be more cost efficient as well as sustainable when was used for PCB recycling (Palanisamy et al., 2022).

5.2.2.2. Epoxy resin recycling. Mechanical recycling (previously explained in section 6.2.1) of the TEG assembly by crushing would result in a mix of composites containing epoxy resin, p-type (PEEK/SWCNTs) and n-type legs (PEEK/SWCNTs/polyethylene glycol). These composites can be used as reinforcement pieces for other materials (Z. Tian et al., 2022), but it is likely that their composition is not uniform, making this application difficult. Another possibility is the application of cryo-shredding followed by a materials separation stage based on their different sizes and/or densities. This process is based on epoxy being quite brittle, while the polymer parts are more flexible and should result in larger shredder sizes.

Another recycling technique is **pyrolysis**. For this technique, the presence of different components in the sample (epoxy resin/PEEK/SWCNTs/polyethylene glycol) should be considered to correctly

estimate the behaviour of the materials during the thermal treatments. The anaerobic (nitrogen atmosphere) and oxidative pyrolysis until 800 °C of epoxy resin reinforced with carbon fibres has been reported in (Ma et al., 2021). The liquid fraction of both types of pyrolysis was composed of methylcyclohexene and phenolic derivatives, whose concentration was lower for the oxidative pyrolysis. The gas fraction from the anaerobic pyrolysis mainly contained CO₂, CH₄, H₂, and H₂O, and from the oxidative pyrolysis CO and CO₂. The pyrolysis of PEEK under inert atmosphere (He) to 900 °C showed two regions: (1) up to 650 °C ether group and ketone group decomposed to phenol and CO₂, and the fluorenone structure moved to the carbonization scheme; (2) until 900 °C the ketone group of fluorenone and from the carbonized structure decomposed to CO₂ (Perng et al., 1999). However, the TGA in inert atmosphere of SWCNT shows the SWCNTs burning at 765 °C (M. Zhang et al., 2002). Thus, the anaerobic pyrolysis of the TEG would result in a liquid fraction made of methylcyclohexene and phenolic derivatives and a gas fraction made of phenol, CO₂, CH₄. As SWCNTs, the most expensive materials in TEGs, would not be recovered this method is unsuitable for TEGs recycling.

Chemical recycling, also known as solvolysis, has been broadly studied for the epoxy resin recycling. This technique comprises the transformation of the resin into monomers, oligomers, or other substances through the addition of solvents under certain reaction

conditions (i.e., residence time, pressure, temperature, catalyst, pulp ratio, agitation). Attention must be paid to the sputtered copper connections, as when using a chemical recycling method, these connections on the surface of the polymer-based strips may also be de-bonded and separated. Chemical recycling of the epoxy resin composites containing resin and carbon fillers could be pursued to depolymerize the resin and release the thermoelectric legs. It is expected that this method does not damage the PEEK due to the difficulties found to depolymerize this molecule. In this way, (Minami et al., 2023; Oliveux et al., 2015) shows that temperatures higher than PEEK melting point (345 °C) are requested for the PEEK solvolysis. (Mu et al., 2022) reports on the use of bond exchange reaction assisted by small molecules in a mixed solvent of dimethylformamide/ethylene glycol (50/50). At a later stage, depolymerized epoxy oligomer (DEO) and the solvents were separated by distillation. It was also demonstrated that this method would be applicable for the recycling carbon fibre reinforced composites (CFRCs) recovering high-value carbon fibre. The decomposition of bisphenol F-type epoxy resin and typical amine curing agent to picric acid was performed using 4 M nitric acid at 80 °C for 30h in (Hanaoka et al., 2021; Minami et al., 2023). Increasing the nitric acid concentration to 7.1M and working at 60 °C M reduce the residence time to 30min (Hanaoka et al., 2022). Epoxy resin can be 99% degraded using supercritical water as shown by (Kim et al., 2019). Working with phenolic resins, phenol and KOH in subcritical water allows a decomposition efficiency of 95.2% (Y. Liu et al., 2012). Super- and subcritical alcohols and mixed systems, such as Methanol, ethanol, n-propanol, iso-propanol, n-butanol, and acetone, and/or KOH as catalyst, can be also used for the epoxy resin degradation (Z. Tian et al., 2022). The highest degradation efficiency using these systems was 97.6%. The use of peracetic acid generated in situ from a mixture of acetic acid and H₂O₂ to oxidize the epoxy resin reached degradation efficiencies of 97% at 65 °C for 5 h (Pérez et al., 2021) achieved total degradation of thermoset epoxy resin using a combination of ionic liquid and alcohol (1-butyl-3-methyl imidazolium chloride and ethylene glycol) at 150 °C in 2.5 h under atmospheric pressure.

Advantages and disadvantages of recycling methods for carbon fiber with epoxy composites are summarized in reference (S. Kumar and Krishnan, 2020). In terms of environmental impact, the chemical solvents can be toxic to the environment in the case of the solvolysis. However, pyrolysis must be performed at higher temperature (higher energy consumption), and the oxidization of the composites produces toxic gases like carbon monoxide (CO) and green housed like carbon dioxide (CO₂). Due to the high energy consumption of pyrolysis, the cost of this technology is much higher compared to solvolysis. Additionally, solvolysis is typically conducted at low temperatures precisely to reduce energy consumption.

5.2.2.3. Reuse of the thermoelectric legs or leg material from TEG. Once the thermoelectric legs have been recovered intact, they may be directly reused in a similar TEG application. This requires that the dissolving of the epoxy embedment could be done in a suitable way without any dimensional, physical or chemical damage of the TE legs.

The thermoelectric composite legs or their shredded granules should be treated by a melt processing step to obtain reusable materials (PEEK/SWCNT composites) with the desired properties. This can be done by melt-compounding, e.g. in an extruder, under addition of the additive polyethylene glycol (if n-type material is aimed) or possibly by adding somehow more SWCNTs to reobtain the initial properties. (Paleo et al., 2022) explains the production process of n-type legs based on SWCNTs and PEEK; accordingly, the recycled PEEK composites could be reused as an input to feed the main hopper of the compounder. This type of treatment has been already done on carbon fibre-reinforced PEEK. The end-of-life polymers were grounded and added to virgin PEEK, conferring to the material even better mechanical properties (Oliveux et al., 2015). However, no references have been found regarding the reuse of

thermoelectric polymers based on CNT/PEEK composites.

5.2.2.4. Recycling of the thermoelectric leg materials. The p- and n-type leg materials could be also recycled to obtain SWCNTs and PEEK separately. However, the decomposition of PEEK requires extreme conditions such as working in sub- and supercritical water system using Na₂CO₃ as catalyst at 703 K during 3 h for reaching a yield of 88% (Tagaya et al., 2004). 2-phenylethanethiolate has been also used as a reagent for an effective depolymerization of PEEK (yield equal to 98%), followed by treatment with organic halides (Minami et al., 2023), but further research must be done on this field for optimizing the reaction conditions and processing times. Moreover, the acid treatment of PEEK becomes very complicated as this polymer is insoluble in all common solvents expects strong acids such as 98% sulphuric acid and hydrofluoric acid (Xu et al., 1992), which are not recommendable due to health hazard, toxicity, corrosive properties, and other potential risks. The thermal treatment was explained above, giving the conclusion of a non-worthy recycling technique.

Table 3 shows a summary of the possible recycling techniques for the TEG:

As a conclusion, the best approach to recycling the TEG is the previous solvolysis of the epoxy resin to obtain TE composite legs (based on PEEK), most likely with nitric acid. Then, reuse of the legs for the fabrication of new ones or other applications, by mixing the recycled composites with virgin PEEK and other fillers if needed. A proposed sequence of techniques for the recycling of TEGs is presented in Fig. 6.

5.2.3. Supercapacitor

The supercapacitor consists of layered polymeric systems. Recovering materials from the SC can be approached in two ways: separating each component individually or treating the SC as a single polymeric system to recover all materials together. Both methods involve mechanical, thermal, and chemical processes, but there may be issues with compatibility, process overlap, and yield efficiency in the latter approach.

5.2.3.1. Separation of the components. Mechanical pre-treatment like trituration can expose components for further processes. However, processing and separating materials from the resulting mix can be challenging. Thus, thermal and/or chemical treatment may be more viable options.

Chemical pre-treatment with solvents can break bonds between printed layers and separate components. However, solvents could have different effects on PET, PANI, and chitosan making further separation of PET infeasible (Y.-B. Zhao et al., 2018). Thermal pre-treatment can remove sealant and separate the top Al/PET layer. Complete degradation of PET, chitosan, PANI, and the current collector adhesive can be achieved at around 500 °C, allowing for easy separation of aluminium.

5.2.3.2. Aluminium and PET recovery from the Al/PET layer. Various recycling techniques for aluminium separation and PET layered polymers have been proposed for waste from beverage and food carton packaging or pharmaceutical blister packaging. These include electro-mechanical separation (Gente et al., 2003) and electrohydraulic fragmentation (Agarwal et al., 2020) although with not very efficient outcomes. More effective chemical methods to leach aluminium using hydrochloric acid, sodium hydroxide or bioleaching with bacteria has been proposed (C. Wang et al., 2015; C. Wang et al., 2015). Solvents can be used to dissolve the polymer, with organic solvent such as benzene-ethanol-water mixture (S. F. Zhang et al., 2014) or with the more environmentally friendly deep eutectic solvents (Nieminen et al., 2020). Phenol has been demonstrated to be able to dissolve waste PET bottle (Ali et al., 2022) as well as Trifluoro acetic acid (TFA) and Trichloro acetic acid (Mahalingam et al., 2015) and Hexafluoro-2-propanol (HFIP) (Pulido et al., 2019). The recovery yield depends on the polymer

Table 3
Summary of the recycling techniques for TEGs.

Components	Composition	Recycling techniques	Recoverable materials	References
Terminals	Copper	Hydrometallurgy: nitric acid 3M at 60 °C	Copper hydroxide	(M. Kumar et al., 2014)
Casing + p-type leg & n-type leg	Epoxy resin, [PEEK/SWCNT] & [PEEK/SWCNT/Polyethylene Glycol]	Mechanical Pyrolysis	Reinforcement for composites Methylcyclohexene and phenolic derivatives, phenol, CO ₂ , CH ₄ with a small amount of C ₂ and C ₃ gases	(Z. Tian et al., 2022) (Ma et al., 2021; Perng et al., 1999; M. Zhang et al., 2002)
Casing	Epoxy resin	HNO ₃ 7.1M/60 °C/30min Supercritical fluid water/150 °C/10min Dimethylformamide/ethylene glycol (50/50)/150 °C/30 min Subcritical water/phenol/KOH/315 °C/30 min Peracetic acid/acetic acid/H ₂ O ₂ /65 °C/5 h 1-butyl-3-methyl imidazolium chloride and ethylene glycol) at 150 °C/2.5 h	Functional chemicals Phenol-like chemicals Depolymerized epoxy oligomer	(Hanaoka et al., 2022; F. Tian et al., 2020) Kim et al. (2019) Mu et al. (2022)
p-type leg & n-type leg	[PEEK/SWCNT] & [PEEK/SWCNT/Polyethylene Glycol]	Mechanical treatment Supercritical water/Na ₂ CO ₃ /430 °C/3 h 2-phenylethanethiolate + treatment with organic halides	Monophenolic, bisphenolic and amine compounds Oxidation products of amine-cured epoxy resin, higher molecular weights compounds Alcohol derivative of the Bisphenol A Dglycidyl ether Crushed PEEK/SWCNTs composites Monomers such as phenol, cresols, and their analogues PEEK monomer	(Y. Liu et al., 2012) Das et al. (2018) Pérez et al. (2021) Oliveux et al. (2015) Tagaya et al. (2004) Minami et al. (2023)

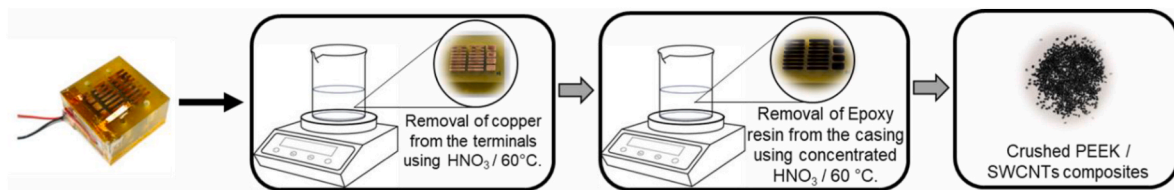


Fig. 6. Sequence of techniques for the recycling of TEGs.

and solvent polarity (Cervantes-Reyes et al., 2015). Other PET depolymerization methods can be used, such as hydrolysis, methanol alcoholysis, ethylene glycol alcoholysis, alcohol-alkali combined depolymerization or ammonolysis (Cao et al., 2022). Another promising chemical technique using sub- and super-critical water can recover all aluminium in solid form without oxidation (Kulkarni et al., 2011). Solvent reusability and impurity removal need to be addressed before implementing these techniques in industry.

These chemicals process need to be assessed for the SC to verify dissolution of other components such as the PANI and Chitosan. (Bhadra et al., 2020) reports that PANI is infusible and insoluble in conventional solvents. PANI usually exists in a powdery form, and forms fine colloidal suspensions rather than being solubilized. Chitosan usually can dissolve in strong hydrochloric acid and weak carboxylic acids like formic acid, acetic acid and propionic acid (Wu and Zhang, 2019), while exhibits low solubility in water and most organic solvents (Li et al., 2019). Talc is practically insoluble in water, dilute mineral acids, and dilute solutions of alkali halides and alkaline hydroxide, while it is soluble in hot concentrated phosphoric acid (Newman et al., 1994).

5.2.3.3. Thermal treatment. Thermal treatment can release valuable materials or separate components for further processes. PET melts above 250 °C, and according to TGA curves, effective loss of mass (up to around 75%) happens between 400 °C and 450 °C (Silva et al., 2018). Chitosan cannot withstand temperatures above 220 °C experiencing up to 90% mass loss at 600 °C (Dey et al., 2016). Crosslinked PANI has a glass transition temperature of around 250 °C with most of mass loss (usually around 80%) at 500 °C (Bao and Liu, 2020; Visakh et al., 2017). The acrylic adhesive should start to degrade above 210 °C, and slightly above this temperature the Al/PET layers could be separated from the rest of the device. This would allow the Al/PET layers to be treated separately as described above, while leaving the rest to undergo further

thermal treatment. In any case a thermal treatment at 500 °C could easily release a clean layer of Aluminium (Yin et al., 2019), but with the loss of PET. High temperature process is usually more costly and more energy demanding than other low temperature process (Gronostajski et al., 2000). Thermal processes like pyrolysis, plasma, or microwave-assisted pyrolysis recover polymeric parts as oil or wax, which can be used as fuel or industrial feedstock (Undri et al., 2014; van Velzen et al., 2020) and eventually employed to support the pyrolysis process. Talc is expected to remain unchanged as shown by the work of (X. Liu et al., 2014) where the decomposition of talc commenced only at ~800 °C, peaking at ~895 °C, with the formation of enstatite and amorphous silica.

5.2.3.4. Carbon and graphite particle recovery. Either after a chemical or thermal treatment, Aluminium is detached and removed, and the PET possibly recovered or decomposed. The solid residue is a mix of talc, carbon particles from the electrode and current collector, and residue from decomposed polymers. Solid residue from PET calcination usually led to a mix of aromatic acids and alkenes compounds (Gornall, 2011; Jia et al., 2020). The challenge is the separation of the two carbon particles. Froth Flotation techniques could be a suitable way to separate the carbon particle from the rest, as demonstrated in a study by (Vasumathi et al., 2023) where graphite is separated from low-grade ores materials. However, froth flotation requires quite large amounts of material to be effective.

Table 4 shows a summary of the possible recycling techniques for the SC:

A proposed sequence of techniques for the recycling of SC is presented in Fig. 7:

Table 4
Potential recycling techniques for the SC.

Component	Composition	Recycling techniques	Recoverable materials	Reference
Whole SC	PET/Al, adhesive, current collector, separator	Mechanical pre-treatment	Mix of crushed composites	(Z. Tian et al., 2022)
		Thermal pre-treatment: 250 °C to break the 3M acrylic adhesive and release the encapsulation layers Thermal pre-treatment: 500 °C	PET/Al layer	Based on 3M adhesive thermal properties ^a
Substrate + encapsulation layer	Aluminium/PET layer	Chemical pre-treatment	Aluminium	Yin et al. (2019)
		The electromechanical separation - previous trituration	Separated SC layers	(Y.-B. Zhao et al., 2018)
		Electrohydraulic fragmentation and separation	De-bonded components: PET - Aluminium	Gente et al. (2003)
		hydrometallurgical processes	De-bonded components: PET - Aluminium	Agarwal et al. (2020)
		Dissolution of PET using solvents and diluents	Aluminium + solid residual (PET)	(C. Wang et al., 2015; C. Wang et al., 2015)
			De-bonded components: PET - Aluminium	Organic solvent (S. F. Zhang et al., 2014); deep eutectic solvents (Nieminen et al., 2020); Phenol (Ali et al., 2022); Trifluoro acetic acid (TFA) and Trichloro acetic acid (Mahalingam et al., 2015); Hexafluoro-2-propanol (HFIP) (Pulido et al., 2019); hydrolysis, methanol alcoholysis, ethylene glycol alcoholysis, alcohol-alkali combined depolymerization or ammonolysis (Cao et al., 2022); sub- and super-critical water (Kulkarni et al., 2011).
Current collector	Activated carbon particle + chitosan + PANI	Froth flotation	Graphite particle	Vasumathi et al. (2023)
Electrodes	Graphite particle + Thermoplastic matrix	Froth flotation	Activated carbon particle	Vasumathi et al. (2023)

^a https://www.3m.com/3M/en_US/p/d/b40071697/.

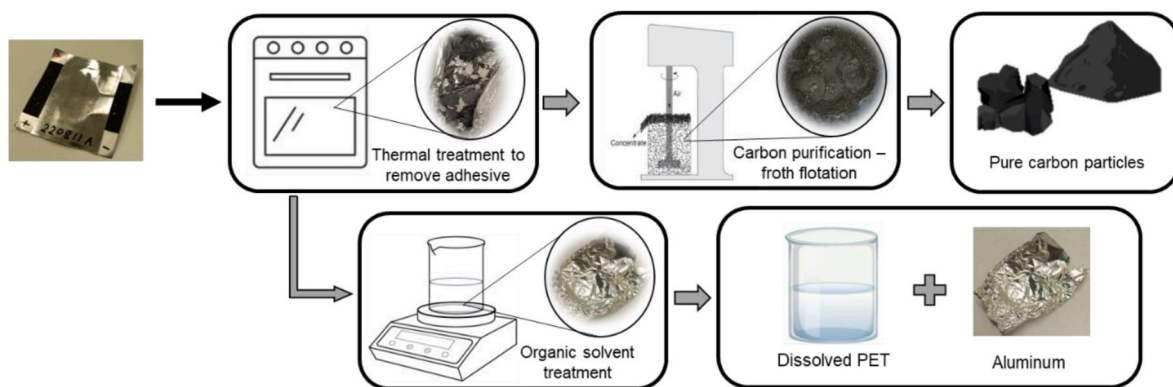


Fig. 7. Sequence of techniques for the recycling of SC. Froth flotation picture extracted from reference (Mesa and Brito-Parada, 2019).

6. Conclusions

The InComEss project, part of the Horizon H2020 program, aims to develop cost-effective EHSs using advanced polymer-based composite materials to power WSN in IoT applications. The TEG is based on PEEK and SWCNT composites, while the PEG is made of PVDF/BYK fibres. In the case of the SC, mainly PET from the substrate and encapsulation layers and Chitosan-PANI/carbon-based composite electrodes. The recyclability of these devices is an important aspect to be evaluated in order to assess the environmental benefits that could result from the recycling of these materials and contribute to a more sustainable EHS system. To assess the recyclability of the devices this article addresses suitable recycling methods for valuable materials and components identifying the recoverable materials from each device. A systematic literature review has been conducted to propose the most suitable recycling techniques.

For the PEG, the thermal and chemical stability of the components makes the definition of methods for their recovery and recycling

intricate. *A priori*, for recycling the encapsulation layer the most suitable methods include microwave assisted degradation of the epoxy composite and pyrolysis of the polyimide. Furthermore, dissolution of the PE film using a green solvent appears to be a viable option. At a later stage, recovery of Ag and Cu would need to be defined according to the results obtained for the rest of the components.

In the case of the TEGs, the main issue comes from the use of PEEK. This polymer exhibits excellent mechanical strength, thermal stability, and chemical resistance. In this way, it becomes very difficult to attack this polymer to deliver the SWCNT, which are the most expensive material within the TEG. For this reason, the best approach seems to be, firstly, the liberation of the thermoelectric legs via solvolysis of the epoxy resin where they are embedded. This can be done with inorganic acid, such as nitric acid at high concentration and temperature. Secondly, the thermoelectric legs can be mechanically treated and used as fillers for PEEK matrices, such as new thermoelectric devices or other applications based on reinforced polymers.

The main materials of interest for recovery in SC are aluminium, PET

and carbon particles. Aluminium, despite its value, may become difficult to recycle in the future development of 3D printed SC due to trend to reduce aluminium foil thickness. PET, a significant component of SCs, has potential for recovery and valorisation. Carbon particles from the electrodes and current collectors can be considered for recovery, although their low weight fraction poses challenges. Various methods for material recovery have been proposed, including mechanical, thermal, and chemical treatments. Chemical treatments involving solvents show promise for separating components, but compatibility issues with PET, PANI, and chitosan polymer must be addressed. Thermal treatments can release valuable materials, and pyrolysis/calcination processes offer the recovery of polymeric parts as fuel or feedstock. The challenge lies in the separation of carbon particles, which could potentially be achieved through froth flotation.

A crucial factor in the recycling of the EHS devices is their end-of-life waste stream, determining if and how they will be collected. As more of these devices find use in real-world applications, it becomes essential to analyse their detachability and explore potential infrastructure for reverse logistics and reparability processes. Next steps envisaged in the InComEss project foresee a comprehensive laboratory assessment of the recovery methods and their applicability to different materials. Adequate techniques must be selected that allow the recycling of individual components without affecting others, and an economic evaluation should be carried out to determine which materials are viably recoverable considering the costs of treatment and the value of the obtained products. Furthermore, a Life Cycle Assessment (LCA) will be performed to evaluate the sustainability of the proposed solution from cradle to grave, including the recycling stages examined in this paper.

CRedit authorship contribution statement

Franco di Persio: Conceptualization, Supervision, Writing – original draft, Writing – review & editing, Data curation, Investigation, Methodology, Validation. **María Blecua:** Writing – original draft, Writing – review & editing, Conceptualization, Validation, Visualization, Investigation, Methodology, Project administration. **Ana Cecilia Chaine:** Writing – original draft, Writing – review & editing, Investigation. **Thomas Daue:** Data curation, Validation. **Cintia Mateo-Mateo:** Data curation, Validation. **Ignacio Ezpeleta:** Data curation, Validation. **Petra Pötschke:** Data curation, Validation. **Beate Krause:** Data curation, Validation. **Ezgi Inci:** Data curation, Validation. **Jürgen Pionteck:** Data curation, Validation. **Timo Punkari:** Data curation, Validation. **Jari Keskinen:** Data curation, Validation. **Matti Mäntysalo:** Data curation, Validation. **Amanda Melo:** Data curation, Validation. **David Esteves:** Data curation, Validation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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List of abbreviations

EHS Energy Harvesting System
PEG Piezoelectric generator

TEG	Thermoelectric generator
SC	Supercapacitor
IoT	Internet of Things
WSN	Wireless sensor network
EU	European Union
PVDF	Polyvinylidene fluoride
SWCNT	Single walled carbon nanotube
PET	Polyethylene terephthalate
FOS	Fibre Optic Sensors
MEMS	Micro Electro-Mechanical System
PEEK	Polyether ether ketone
PANI	Polyaniline
BOM	Bill of Material
TE	Thermoelectric
PE	Piezoelectric
PP	Polypropylene
APS	Ammonium persulfate
WEEE	Waste Electrical and Electronic Equipment
TA	Tartaric acid
NMP	N-methyl pyrrolidone
LIB	Lithium-Ion Batteries
DMF	Dimethylformamide
DMP	Dimethyl phthalate
THF	Tetrahydrofuran
FAMES	Fatty acid methyl esters
DMSO	Dimethyl sulfoxide
HF	Hydrogen fluoride
Pis	Polyimides
DEO	Depolymerized epoxy oligomer
CFRCs	Carbon Fibre Reinforced Composites
TFA	Trifluoro acetic acid
HFIP	Hexafluoro-2-propanol

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